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Single Reinhibition Additive Package For Use With Certified Military Antifreeze Recycling Systems

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To decrease the cost of recycling Military Specification antifreeze MIL-A-46153, several prototype antifreeze additive formulations are examined with two (2) previously approved commercial recycling systems to determine a single reinhibition additive package which can be used with both recycling systems. Prior to this investigation each of the approved recycling systems employed proprietary additive packages which invariably add to the cost of recycling used military antifreeze.

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I. INTRODUCTION

Background

In August 1993 the U.S. Army Belvoir Research, Development, and Engineering Center (BRDEC), now the Mobility Technology Center-Belvoir (MTC-B), completed its investigation of commercial antifreeze recycling systems. The results are available in U.S. Army Tank-Automotive Research, Development, and Engineering Center (TARDEC) Technical Report TR-13638[1]. As a consequence of that study, two (2) commercial recycling systems were approved for use with Military Specification MIL-A-46153[2] (Antifreeze, Ethylene Glycol, Inhibited, Heavy Duty, Single Package) and are currently being used in the field by military personnel[3]. The systems included the Finish-Thompson Inc (FTI), BE Series system and the BG Products Inc (BG), Cool'r Clean'r system.

Both systems are on-site, portable systems which are operated by the user. After contaminates are removed, each system employs a proprietary additive package to restore or reinhibit the used antifreeze to an acceptable level of performance permitting its reuse. The BG and FTI proprietary additive packages employ similar additives but are formulated distinctively to maximize their performance in the individual systems. For example, the BG system produces a single solution of purified water and ethylene glycol (EG). The BG additive package is formulated to be added directly to this solution. The FTI system separates the water and EG and produces two (2) purified solutions. The FTI packaged is formulated to be added to the EG first and then combined with the recovered water.

In addition to the dissimilar additive packages, the methods of recycling are different. The BG system uses ion exchange to remove contaminants whereas, the FTI system uses vacuum distillation. Ion exchange utilizes chemically reactive resins to remove contaminates and clean used antifreeze[4]. Distillation employs a combination of heat, evaporation, and condensation to purify spent antifreeze[5]. Each method affects the chemistry of the resultant, uninhibited antifreeze and how the uninhibited antifreeze will react to the subsequent additive package, making it difficult to interchange the additive packages between the two systems.

Objective

To increase the cost savings of recycling MIL-A-46153, MTC-B began this study to develop a single reinhibition additive package (SRP) that would be completely interchangeable with present military approved commercial recycling systems. For example, the use of a military SRP detail specification could reduce the cost of the

additive package compared to current cost of the individual FTI and BG additive packages. Appendix Table 1 shows the suggested possible savings based on employing Military Specification MIL-A-53009[6] (Additive, Antifreeze Extender Liquid Cooling Systems) for antifreeze reinhibition. MIL-A-53009 or a SRP detail specification similar to MIL-A-53009 may offer comparable cost savings. In addition to the expected cost savings, some organizations may find it logistically easier to manage one container of additive instead of the two required for the two-part additive packages of FTI and BG recycling systems.

II. INVESTIGATION

Approach

Initial compatibility examinations of the FTI and BG packages indicated complete chemical incompatibility between the systems and additives as indicated by formation of moderate amounts of precipitate for those combinations listed in appendix Table 2. The compatibility test involved admixing combinations of each and allowing separate samples of the same mixture to sit over night at room temperature and in an oven set at 60°C. For each sample, the precipitate appeared flocculent in texture and yellow-white to brown in color. Although some mixture combinations gave pH and RA values (see appendix Table 3) comparable to the standard solutions, these samples nevertheless formed significant amounts of precipitate. Hence the need for a single SRP for use with recycling was established.

Several prototype SRP formulations were prepared and subjected to laboratory testing. These formulations were based on the current MIL-A-53009 antifreeze extender additive formula and information obtained from various literature sources concerning corrosion inhibitors for ethylene glycol (EG) and propylene glycol (PG) antifreezes.[7,8,9,10,11,12,13,14] The prototype formulations, including the MIL-A-53009 formulation are given in appendix Tables 4a-4b.

Preparation of the SRP prototype formulations began with initially dissolving the formula's component with the largest concentration in the base fluid. For example, for SRP#1 sodium metaborate was added to the distilled water with continuous stirring at temperatures between 30°C and 45°C until the solution became clear. For the formulations SRP#3 and SRP#6 containing ethylene glycol (EG), the EG was considered the base fluid. The heating of the glycol containing formulations ranged from 55°C to 60°C. For both the EG and water base formulations, the remaining components were added sequentially with continuous stirring and temperatures ten (10) to fifteen (15) degrees lower than the starting temperature.

SRP formulations were examined for effectiveness in recycled, uninhibited antifreeze only if the resultant solution did not produce a precipitate or a large phase separation (e.g., non-homogeneous layer). The examination included ASTM standard laboratory antifreeze tests[15] shown in appendix Table 5 and a in-house storage stability

test. The ASTM performance tests were chosen to determine general corrosion protection abilities (D1384) and aluminum alloy corrosion protection abilities under heat-rejecting conditions (D4340) for the SRP's. The latter test, D4340, is not normally employed for heavy duty antifreeze applications because of the limited number of aluminum components found in most conventional, large diesel engines. However, because of an increasing trend[16] by engine manufacturers to use more aluminum in large engines to reduce weight and improve engine efficiency, D4340 was conducted. The storage stability test consisted of placing 100 milliliters (mL) of the SRP in a stoppered graduated cylinder and allowing the sample to stand over night at room temperature (≈22°C). After examining the solution the following morning, the same unstoppered sample was placed in an oven set at a temperature of 60°C for 24 hours, removed, and examined again. Observation of excessive amounts of precipitate or a large phase change constituted an unstable formulation and testing of that specific formulation was discontinued.

SRP formulations passing the preliminary storage test were then examined for pH and buffer capacity (i.e., reserve alkalinity (RA)). If the results of the pH and RA tests were considered acceptable, the SRP formulation was then examined further in uninhibited, recycled antifreeze from the BG system. Three (3) and six (6) SRP volume percent of uninhibited antifreeze were employed as dosage rates. These rates were determined as the most effective and convenient based on MTC-B's experience with MIL-A-53009. In addition, similar rates are prescribed for the proprietary additive packages of BG and FTI systems. SRP formulations giving acceptable results in BG uninhibited recycled antifreeze were then examined in FTI uninhibited, recycled antifreeze. In addition to examining MTC-B prototype formulations with the BG and FTI systems, MIL-A-53009 was also examined as a possible SRP candidate. MIL-A-53009 was chosen as a feasible candidate because of its past record as a service life extender for used MIL-A-46153.

For the preparation of the uninhibited recycled antifreeze, one (1) common feedstock of used antifreeze was employed with both recycling systems. The used antifreeze was prepared from a mixture of deteriorated Military Specification MIL-A-11755[17] (Antifreeze, Arctic-Type) for arctic weather antifreeze in which the copper inhibitor (e.g., sodium mercaptobenzothiazole) had precipitated out of solution and a used MIL-A-46153 collected from a field source. After recycling the used antifreeze through each system, the resultant recycled antifreeze was adjusted so its freeze point, as tested by ASTM method D3321 in appendix Table 5, was approximately -20°F (≈45% glycol by volume) when inhibited with the subsequent SRP. This was done so that the SRP formulations would encounter similar glycol/water concentrations and react uniformly in the uninhibited antifreeze from each recycling system.

Experimental

The SRP#1 formulation formed a white precipitate during the room temperature phase of the storage test and was not tested further. SRP#2 formed small amounts of a white, flocculent precipitate during the oven phase of the storage test. The precipitate was suspended throughout the solution and was initially attributed to water evaporation

and not actual chemical instability. However, upon examining SRP#2 further for its heat-rejecting aluminum corrosion protection abilities (ASTM D4340) in BG uninhibited recycled antifreeze, a 3% concentration sample failed D4340 with a corrosion rate of 8.8mg/cm^2/week. The testing of SRP#2 was discontinued.

The SRP#3 and SRP#4 formulations produced no precipitate or phase change after being prepared. However, upon adding SRP#3 and SRP#4 to the BG uninhibited recycled antifreeze, the RA's was extremely low. For example, the 3% and 6% concentration solutions with SRP#3 had RA's of 2.3mL and 4.4mL respectively. For SRP#4, the 3% and 6% concentration solutions had RA's of 1.1mL and 2.8mL respectively. ASTM recommends however that a prediluted heavy duty antifreeze, having a 50% by volume EG minimum[18], should have a RA of a least 5mL to assure acceptable performance. Although the solutions were approximately only 45% EG, the RA's were not expected to increase with additional EG present.

To determine the possible effects of the low RA, SRP#4 blended into uninhibited, recycled BG and FTI antifreeze was subjected to the heat-rejecting aluminum test (D4340) and the glassware corrosion test (D1384). The results are shown in appendix Tables 6 and 7. For the D4340, the 3% SRP#4 concentration solution exceeded the ASTM recommended corrosion rate of 1.0mg/cm^2/week and was therefore eliminated from further testing. The 6% SRP#4 concentration in BG uninhibited, recycled antifreeze was below the ASTM rate and the aluminum coupon appeared clean and unmarked after the test was completed. However, the SRP#4 6% concentration in FTI uninhibited, recycled antifreeze failed D4340 with a corrosion rate of 5.8mg/cm^2/week. For D1384, SRP#4 in FTI met ASTM recommendations. The SRP#4 in BG solution failed D1384 with excessive corrosion of the aluminum coupon. Because of the low RA values and the performance inconsistencies observed for SRP#4 in the FTI and BG uninhibited, recycled antifreezes, testing of SRP#3 and SRP#4 was discontinued. As with SRP#3 and SRP#4, the SRP#8 formulation also produced low RA values when mixed with BG uninhibited, recycled antifreeze. The RA values for the 3% and 6% concentration solutions were 2.3mL and 3.8mL respectively. Because of the low RA values, further testing of SRP#8 was discontinued.

Formulations SRP#5 and SRP#6 formed precipitates during the SRP preparation procedure. For SRP#5, the borax would not completely dissolve in the heated EG. For the SRP#6 formulation, potassium silicate remained insoluble after several minutes of agitation. Examination of the SRP#5 and SRP#6 formulations was discontinued.

The concentrate solutions of SRP#7 and SRP#9 were clear and bright after their preparation and storage tests. Along with their initial stability, SRP#7 and SRP#9 pH and RA values in BG uninhibited recycled antifreeze were favorable at the 3% and 6% concentration levels (see appendix Table 8). The pH and RA results were found to be acceptable in FTI uninhibited recycled antifreeze as well, as shown in appendix Table 8. MIL-A-53009 also produced similar results in both BG and FTI solutions as shown in appendix Table 8. Performance examination of SRP#7, SRP#9, and MIL-A-53009

results are shown in appendix Tables 6 and 7. For the BG sample's D4340 and D1384 test results, corrosion protection appeared slightly maximized with the larger inhibitor levels of the 6% versus the 3% concentration solutions. As such, only 6% concentrations were tested with the FTI samples. Although some samples passed individual tests, neither of the SRP formulations nor the MIL-A-53009 were able to pass both tests with each recycling system uninhibited, recycled antifreeze.

Results and Discussion

The inconsistencies of performance and quality exhibited by the various combinations of SRP formulations and MIL-A-53009 confirm the difficulty of designing a SRP for two different recycling systems. MIL-A-53009 was fairly effective for general corrosion protection but lacked specificity for protecting against aluminum corrosion. The SRP#7 and SRP#9 formulations were unable to successfully pass D1384 when admixed in both uninhibited recycled base antifreezes. These discrepancies in performance are attributed to the differences in the recycling processes and the recycled product they each generate. Despite the discovered inherent chemistry differences between the BG and FTI recycled antifreeze, a single reinhibition formulation appears to be possible from a combination of the MIL-A-53009, SRP#7 and SRP#9 formulations shown in appendix Tables 4a-4b. To produce the SRP additional time and tests will be required to determine the exact formulation.

III. CONCLUSION

As observed during the initial study results, the interchangeability of the current BG and FTI proprietary reinhibition packages is not possible with used MIL-A-46153. Each recycling system affects the chemistry of the resultant, uninhibited antifreeze differently, thereby making it difficult to interchange the additive packages between the two systems. In addition, used MIL-A-46153 past studies[19,20] have shown to detrimentally affect the reinhibition steps of some recycling systems because of the residual borax content in used MIL-A-46153. This effect was observed in previous studies as exhibited by the resultant recycled antifreeze as excessive precipitates and abnormal RA and pH values. Of the two systems evaluated the BG system has shown the least affect of the MIL-A-46153 borax effect. The FTI system was affected such that a special additive package had to be designed for use with MIL-A-46153. For this study the residual borax effect undoubtedly contributed to the performance inconsistencies of the BG, FTI, and prototype additive packages examined (i.e., causing difficulty for a prospective SRP to react and perform similarly in uninhibited antifreeze from both the BG and FTI systems).

The results of this study indicated that a single reinhibition package is possible with MIL-A-46153, however additional work is required to finalize a formulation. Due to the Department of Defense's (DOD) desire to incorporate more commercial standards, additional work which would result in detailed SRP specification would be difficult to justify and fund. More importantly an effort to develop a standard has begun which will allow the authorized use of commercial antifreeze in all military vehicles.

IV. RECOMMENDATIONS

As a viable extension of this work, a second study covering the development of a commercial SRP standard is recommended. Given that no less than three (3) major manufacturers (i.e., Fleetguard, Grace Dearborn, and Penray) presently produce additive reinhibition packages for the majority of antifreeze recyclers currently on the market, it would be relatively easy to develop a multisystem product. In addition, the use of commercial antifreeze would eliminate the borax effect associated with MIL-A-46153 and further reduce the difficulty of developing a multisystem additive. For example, the borax levels of most U.S. antifreezes compared to MIL-A-46153's 4% borax by weight has ranged from 0% to 2%[21,22,23,24] As with MIL-A-46153, a commercial SRP would be expected to decrease the cost of recycling antifreeze.

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TABLE 1
Cost Comparison of Proprietary Recycled Antifreeze Additive Packages and MIL-A-53009

	MIL-A-53009	FTI, REMILFTH-55 Part A & Part B	BG, BG570A & BG570B
Unit/Price[1]	1qt/$2.65	Part A = 55gal/$1650.00 Part B = 55gal/$1650.00	BG570A = 55gal/$1755.67 BG570B = 55gal/$2032.37
Cost/gal of inhibitor(s)	$10.60	$60.00[2]	$68.88[3]
Cost/ 500gal of used 50/50 antifreeze	$318.00	$450.00	$1033.10

Calculations:
Cost/gal of MIL-A-53009 = 1qt/$2.65 x 4qt/1gal
MIL-A-53009 cost/500gal of used 50/50 antifreeze = (500 gal of recycled uninhibited water/glycol soln') x (6% dosage rate[4] x cost/gal of MIL-A-53009)

FTI, Cost/gal of Part A = $1650 ÷ 55gal,
Cost/gal of Part B = $1650 ÷ 55gal
FTI, Cost/500gal of used 50/50 antifreeze = {(250 gal of recycled uninhibited glycol soln') x (3% dosage rate[5] x cost/gal of Part A)} + {(250 gal of recycled uninhibited glycol soln') x (3% dosage rate x Cost/gal of Part B)}

BG, Cost/gal of BG570 A = $1755.67 ÷ 55gal,
Cost/gal of BG570 B = $2032.37 ÷ 55gal
BG, Cost/500gal of used 50/50 antifreeze = {(500 gal of recycled uninhibited water/ glycol soln') x (3% dosage rate[6] x cost/gal of BG570A)} + {(500 gal of recycled uninhibited water/glycol soln') x (3% dosage rate x Cost/gal of BG570B)}

[1] As quoted by the Defense General Supply Center 30 Aug 1995 and 30 Oct 1995.
[2] Combined cost of part A and part B.
[3] Combined cost of pBG70A and BG570B.
[4] 6% by total volume of uninhibited, recycled water/ethylene glycol solution.
[5] 3% by total volume of uninhibited, recycled ethylene glycol solution.
[6] 3% by total volume of uninhibited, recycled water/ethylene glycol solution.

TABLE 2.
Proprietary Inhibitor Compatibility Test Results

Incompatible Packages	Compatible Packages
1. BG Recycled + 3% FTI MIL Additive[1]	1. BG Recycled + 3% BG HD Additive[5]
2. BG Recycled + 6% FTI MIL Additive	2. FTI Recycled Conc. + 3% FTI MIL Additive[6]
3. BG Recycled + 3% of EG conc[2]., FTI MIL Add.	
4. FTI Recycled EG Conc. + 3% BG HD Additive[3]	
5. FTI Recycled EG Conc. + 6% BG HD Additive	
6. FTI Recycled EG/Water Soln'[4]. + 3% BG HD Add.	
7. FTI Recycled EG/Water Soln' + 6% BG HD Add.	

[1] FTI Product = REMILFTH-55
[2] 6FTI recovered EG reinhibited prior to mixing with recovered water.
[3] BG Product = BG570A and BG570B (Heavy Duty).
[4] FTI recovered EG and recovered water mixed prior to reinhibition.
[5] Standard BG sample mixture.
[6] Standard FTI sample mixture.

TABLE 3.
pH, Reserve Alkalinity (RA), and Freeze Point (FP) Values for Intermixed Proprietary Packages

Sample	pH	RA	FP
BG Recycled + 3% BG HD Additive[1]	11.1	6.1mL	-22°F
BG Recycled + 3% FTI MIL Additive	12.5	20.9mL	-25°F
BG Recycled + 6% FTI MIL Additive	13.5	37.5mL	-30°F
BG Recycled + 3% of EG conc., FTI MIL Add.	12.6	8.6mL	-23°F
FTI Recycled Conc. + 3% FTI MIL Additive[2]	12.6	9.9mL	-25°F
FTI Recycled EG Conc. + 3% BG HD Additive	7.9	2.6mL	-25°F
FTI Recycled EG Conc. + 6% BG HD Additive	8.6	5.3mL	-20°F
FTI Recycled EG/Water + 3% BG HD Additive	8.6	5.5mL	-20°F
FTI Recycled EG/Water + 6% BG HD Additive	10.9	11.0mL	-18°F

[1] Standard BG sample mixture.
[2] Standard FTI sample mixture.

TABLE 4a.
Single Reinhibition Package (SRP) Prototype Formulations
weight percent

	MIL-A-53009	SRP#1	SRP#2	SRP#3	SRP#4	SRP#5
Sodium Metaborate	29%	15%	29%	0%	0%	0%
Borax	0%	0%	0%	10%	0%	15%
Sodium Nitrite	0%	3%	3%	3%	1%	4%
Sodium Nitrate	0%	3%	3%	1.5%	2%	2%
Sodium Tolytriazole	0%	3%	3%	3%	2%	3%
Sodium Mercaptobenzothiazole	3%	0%	0%	0%	0%	0%
Sodium Phosphate, Dibasic	0%	0%	0%	0%	6%	0%
Sodium Molybdate	0%	0%	0%	0%	3%	0%
Sodium Silicate	0%	0%	0%	0%	0%	0%
Potassium Silicate Soln'	4.6%	3%	3%	1.5	3%	2%
Silicate Stabilizer	0%	0%	0.6%	0.6%	0.6%	0.4%
Distilled Water	63.4%	73%	58.4%	32.2%	82.4%	0%
Ethylene Glycol	0%	0%	0%	48.2%	0%	73.6%
pH of Concentrate	12.5	nt[1]	12.4	7.9	10.3	nt
RA of Concentrate	>40mL	nt	>40mL	>40mL	>40mL	nt

[1] nt = not rated

TABLE 4b.
Single Reinhibition Package (SRP) Prototype Formulations (cont.)
weight percent

	SRP #6	SRP#7	SRP#8	SRP#9
Sodium Metaborate	0%	20%	0%	32%
Borax	0%	0%	0%	0%
Sodium Nitrite	1%	4%	2%	4%
Sodium Nitrate	1%	2%	2%	3%
Sodium Tolytriazole	2%	2.3%	2%	4%
Sodium Mercaptobenzothiazole	0%	0%	0%	0%
Sodium Phosphate, Dibasic	6%	0%	10%	0%
Sodium Molybdate	3%	0%	5%	0%
Sodium Silicate	0%	0%	0%	2%
Potassium Silicate Soln'	3%	2%	3%	0%
Silicate Stabilizer	0.6%	0.6%	0.6%	0.6%
Distilled Water	33.4%	69.1%	75.4%	54.4%
Ethylene Glycol	50.0%	0%	0%	0%
pH of Concentrate	nt	11.6	10.6	13.1
RA of Concentrate	nt	>40mL	>40mL	>40mL

TABLE 5.
Laboratory Tests

Performance Tests

- Corrosion Test for Engine Coolants in Glassware (ASTM D-1384)
- Corrosion of Cast Aluminum Alloys in Engine Coolants Under Heat=-Rejecting Conditions (ASTM D-4340)

Quality Tests

- Reserve Alkalinity (RA) of Engine Antifreeze, Antirusts, and Coolants (ASTM D-1121)
- pH of Engine Antifreezes, Antirusts, and Coolants (ASTM D-1287)
- Use of The Refractometer for Determining the Freezing Point of Aqueous Engine Coolants (ASTM D-3321)

Storage Stability Test

- In-House Storage Stability Test (non-ASTM)

TABLE 6.
Heat-rejecting Aluminum Surface Test, ASTM D4340

Sample	Corrosion Rate	Before Test pH/RA	After Test pH/RA
3% SRP#4 in BG	4.9mg/cm²/wk	9.7/0.8mL	8.9/0.4mL
6% SRP#4 in BG	0.5mg/cm²wk	9.4/0.7mL	9.4/0.6mL
6% SRP#4 in FTI	5.8mg/cm²/wk	7.8/1.0mL	7.7/1.1mL
6% SRP#7 in BG	2.8mg/cm²/wk	10.3/3.5mL	9.6/3.3mL
6% SRP#7 in FTI	0.0mg/cm²/wk	9.1/6.7mL	9.0/6.2mL
3% SRP#9 in BG	7.2mg/cm²/wk	10.2/2.8mL	9.6/2.6mL
6% SRP#9 in BG	9.6mg/cm²/wk	10.4/9.3mL	10.0/9.8mL
6% SRP#9 in FTI	1.6mg/cm²/wk	9.4/11.5mL	9.2/11.7mL
3% 53009 in BG	5.7mg/cm²/wk	9.8/4.6mL	8.8/3.8mL
6% 53009 in BG	6.8mg/cm²/wk	10.7/11.1mL	9.3/10.6mL
6% 53009 in FTI	4.3mg/cm²/wk	8.9/9.3mL	8.8/9.3mL
ASTM limits, max.	1.0mg/cm²/wk	nr[1]	nt[2]

[1] nr = not reported
[2] nt = not tested

TABLE 7.
Glassware Corrosion Test, ASTM D1384
weight change, mg/coupon[1]

Sample	Copper	Solder	Brass	Steel	Iron	Aluminum
6% SRP#4 in BG	1	2	2	+1	+1	64
6% SRP#4 in FTI	0	4	2	1	1	22
6% SRP#7 in BG	+1	6	0	0	+1	6
6% SRP#7 in FTI	0	18	1	0	+2	53
3% SRP#9 in BG	0	8	1	0	0	24
6% SRP#9 in BG	1	16	2	1	+1	11
6% SRP#9 in FTI	0	13	1	0	+2	61
3% 53009 in BG	+1	2	+2	1	4	4
6% 53009 in BG	1	1	+1	+1	+1	+1
6% 53009 in FTI	0	0	0	+1	+2	27
ASTM limits, max.	10	30	10	10	10	30

[1] The changes are weight losses except "+" indicates a weight gain.

TABLE 8.
pH and RA Values for SRP#7, SRP#9, and MIL-A-53009

Sample	pH	RA
3% SRP#7 in BG	10.0	6.7mL
6% SRP#7 in BG	10.3	14.3mL
3% SRP#7 in FTI	8.1	5.7mL
6% SRP#7 in FTI	8.6	12.2mL
3% SRP#9 in BG	8.4	9.6mL
6% SRP#9 in BG	8.9	20.6mL
3% SRP#9 in FTI	10.0	12.2mL
6% SRP#9 in FTI	10.3	23.8mL
3% 53009 in BG	9.0	9.9mL
6% 53009 in BG	10.3	17.6mL
3% 53009 in FTI	8.1	8.2mL
6% 53009 in FTI	8.8	16.8mL